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Research Article

Synthesis and Characterization of Copoly(Anethole-Stearyl Acrylate) as Phenol Adsorbent

Desi Suci Handayani*, Edi Pramono, Anita Kusuma Dewi

Department of Chemistry, Faculty of Mathematics and Natural Sciences, Sebelas Maret University, Surakarta 57126, Indonesia

**Email:* *desisuci@staff.uns.ac.id*

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Abstract

This study aims to synthesize copoly (Anethole-stearyl acrylate, SA) through cationic polymerization at room temperature (28–36°C) under a nitrogen atmosphere. The synthesis was carried out without a solvent using $BF_3O(C_2H_5)_2$ as the initiator. SA was added in varying weights of 2%, 4%, and 6% relative to anethole. The product obtained from the synthesis was then applied as a phenol adsorbent using the batch method at different contact times. FTIR (Fourier Transform Infrared) analysis showed that methoxy group ($-OCH_3$) termination occurred at wavenumber 1147 cm^{-1}, followed by the loss of vinyl group (C=C) at 1633–1654 cm^{-1}. In addition, the loss of =C-H vinyl bending and stretching group absorption occurred at 964–998 cm^{-1} and 3025–3095 cm^{-1}, respectively. Structural analysis using ^{1}H-NMR showed the loss of the vinyl group proton signal at a chemical shift (δH) of 5.8–39 ppm. The addition of 2%, 4%, and 6% SA led to an increase in intrinsic viscosity of 26.891, 41.093, and 55.336, respectively. The morphology of copoly showed the presence of various cavities. The degradation temperature of the product increased with the addition of SA. During the application of copoly as a phenol adsorbent, the 6% (w/w) concentration had the largest adsorption capacity of 2.22 mg/g.

Keywords: Adsorbent, anethole, cationic copolymerization, phenol, stearyl acrylate

1. INTRODUCTION

Anethole (1-methoxy-4-(1-propenyl)benzene), with the molecular formula $C_{10}H_{12}O$, is an aromatic compound found in the essential oil of the fennel plant. The compound also accounts for 80–90%, 90%, and 80% of the essential oil found in anise, star anise, and fennel [1]. Several studies have shown that anethole has two major isomers, namely cis and trans. The trans isomer is more widely used due to its relatively high abundance and non-toxic nature compared to the cis isomer [2].

Anethole can be subjected to various modifications to produce commonly used chemicals, such as p-methoxy benzaldehyde and anisic acid. The presence of allyl and methoxy groups also facilitates the production of various polymeric materials [3,4]. Despite its potential, polymerization typically changes the morphology of anethole, leading to the presence of a grainy structure [5]. To change the morphology, it can be copolymerized with stearyl acrylate (SA) to produce a polymer with a hollow structure. This is because SA has a sponge-like structure [6,7], which provides space for adsorption [8,9].

According to previous studies, the increase in water pollution due to organic waste and demand for clean water are serious problems, affecting various countries. Organic waste containing phenol is often dangerous because phenolic compounds are toxic, corrosive, carcinogenic, and difficult to degrade [10]. To address these problems, a polymer adsorbent has been reported to be an alternative for replacing activated carbon in removing pollutants. It is assumed that hydrophobic interactions, hydrogen bonds, and π-π interactions, generated by the accumulation of benzene rings on phenol molecules and the adsorbent, play a role in the adsorption process. Several polymers have been used as adsorbents for organic waste containing phenols, including macroporous copoly (methyl methacrylate-DVB), copoly (styrene-DVB), copoly (Np vinylbenzylacetamide)[11], polystyrene, polyacrylate esters, caropoly copolymer styrene, and polymethyl methacrylate[12], however, their adsorption capacity and selectivity toward phenol are relatively low. In this study, the synthesis of copoly(Anethole–SA) was carried out because previous approaches had not achieved optimal performance in phenol adsorption. The combination of the aromatic groups from anethole and the polar functional groups from stearyl acrylate is expected to yield an adsorbent with high adsorption capacity, enhanced selectivity, and excellent chemical stability. Due to its aromatic structure and active π–π stacking capability, anethole can interact with the aromatic ring of phenol via π–π interactions. Conversely, stearyl acrylate (SA) contains –COOH and –OH groups that enable hydrogen bonding with the –OH group of phenol.

2. RESEARCH METHODS

Instrument and Materials

The instruments used were a set of reflux equipment, Mettler PB 3000 Type ER-182 A Balance, Ostwald Viscometer (Iwaki Pyrex), FTIR Spectrophotometer (Fourier Transform Infrared, Shimadzu Type FTIR-8201 PC KBr), ^{1}H-NMR Spectrophotometer (Agilent 500 MHz), SEM-EDX (Scanning Electron Microscopy-Energy Dispersive X-ray, FEI Quanta 250), UV- Vis (Hitachi UH5300), and Thermal Analyzer (Linseis type PT 1600).

All materials used had E. Merck specifications, except for those specifically mentioned: SA (Aldrich), nitrogen gas (PT. Samator), aquades (Integrated Laboratory, Faculty of Mathematics and Natural Sciences, Sebelas Maret University), silica gel (Brataco).

Synthesis of Copoly(Anethole-SA)

The synthesis of copoly was based on the study by Handayani[13]. The copolymerization process was carried out by flowing nitrogen gas at room temperature. Approximately 2 g of anethole was mixed with SA in weight variations of 2%, 4%, and 6%. Subsequently, the two monomers were stirred with a magnetic stirrer for 1 hour, and the copolymerization was carried out for 4 hours. Every hour, 0.05 mL of $BF_3O(C_2H_5)_2$ was added as initiator. The process was stopped (termination stage) by adding 0.1 mL of methanol. The products obtained were dissolved in chloroform and neutralized by liquid-liquid extraction with distilled water until the pH was neutral. The organic phase was dried with anhydrous Na_2SO_4 and evaporated at room temperature. The product was dried, analyzed, and characterized using an FTIR spectrophotometer, ^{1}H-NMR, Ostwald viscosimeter, SEM-EDX, and TGA (Thermogravimetric Analysis). Yield was calculated based on Equation 1.

$$\%\,Yield = \frac{Products\ obtained}{Theoretical\ product}\ x\ 100 \qquad (1)$$

Copoly(Anethole-SA) as Phenol Adsorbent

The adsorption experiment was conducted at room temperature by adding 100 mg of adsorbent to 25 mL of phenol solution, with a different initial concentration of 10 mg.L^{-1}. The mixture was stirred at a speed of 250 rpm with time variations of 1 to 5 hours. The adsorption process was repeated 2 times (duplo). The concentration of phenol was analyzed using a UV-Vis spectrophotometer. The absorbance was measured according to the maximum wavelength (269.5 nm), and the adsorption capacity (q) was calculated based on Equation 2.

$$q = \frac{V(C_0 - C_t)}{m} \qquad (2)$$

Where q denoted the adsorption capacity (mg/g), C_0 was the initial concentration of phenol (mg/L), C_t was the final concentration of phenol after being adsorbed (mg/L), V was the solution volume (L), and m was the mass of adsorbent (g).

3. RESULTS AND DISCUSSION

The $BF_3O(C_2H_5)_2$ initiator forms a coordination bond by sharing a lone pair of electrons from the O atom to the B atom. As a result, the -C_2H_5 group tends to become positively charged (a carbonium ion) and can undergo a polymerization reaction by adding monomers to form new carbonium ions[14]. The initiator was added gradually, which allowed the process to occur repeatedly, yielding copolymers with long chains. The formation of $BF_3O(C_2H_5)_2$ initiator is shown in **Figure 1**.

Figure 1. Formation of the $BF_3O(C_2H_5)_2$ Initiator

The synthesis process was carried out in a nitrogen atmosphere to protect the copolymerization reaction from water vapor or other proton-donating molecules that could interfere with the cationic polymerization process. Moreover, water and alcohol cause the initiator to become inactive[14]. The anethole monomer is initiated first due to the higher mass ratio[14] and lower steric hindrance compared to SA[15]. The possible initiation reactions between Anethole, SA, and $BF_3O(C_2H_5)_2$ initiator are displayed in **Figures 2** and **3**.

The polymer chain can undergo elongation by forming covalent bonds [16] between anethole and SA, which is initiated. One of the possible reactions is called the propagation step, as shown in **Figure 4**. The propagation step affects polymer chain length and relative molecular mass [16]. Furthermore, it is influenced by the stability of the carbocation in the monomer [8]. One of the possible terminations of copoly is shown in **Figure 5**. The synthesis of copoly produced yields and physical forms as shown in **Table 1** and **Figure 6**. The addition of stearyl acrylate (SA) influences the color of the copolymer, with increasing SA content causing the color to shift from white to yellowish.

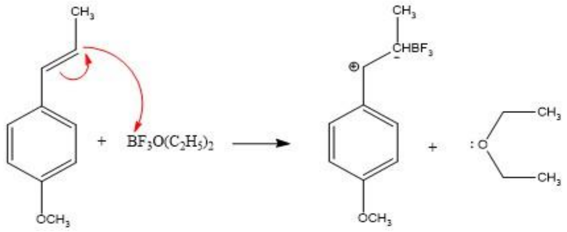

Figure 2. Possible Initiation between Anethole with $BF_3O(C_2H_5)_2$

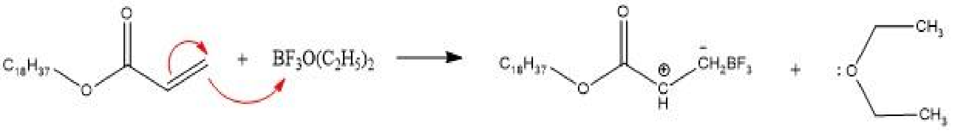

Figure 3. Possible Initiation between SA with $BF_3O(C_2H_5)_2$

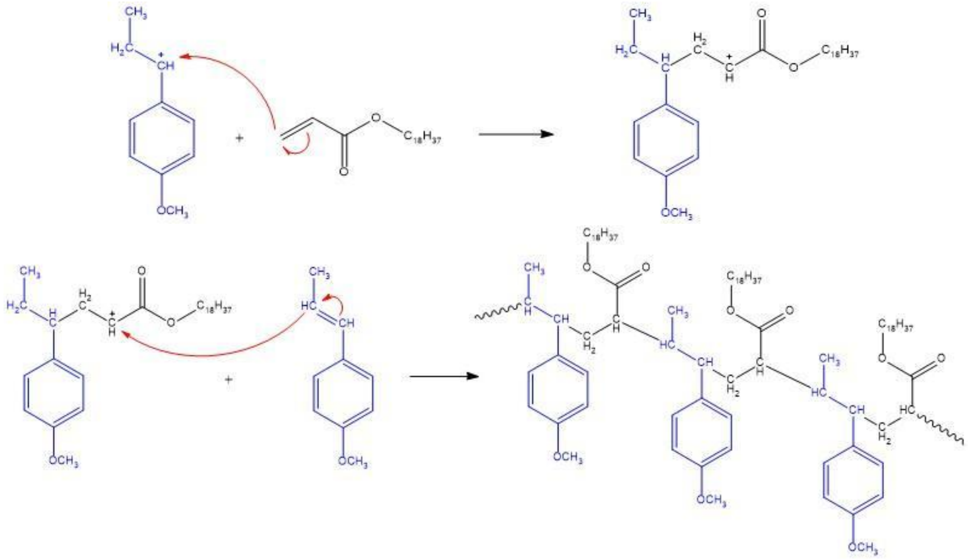

Figure 4. The possibility of Propagation between Anethole and SA has been initiated

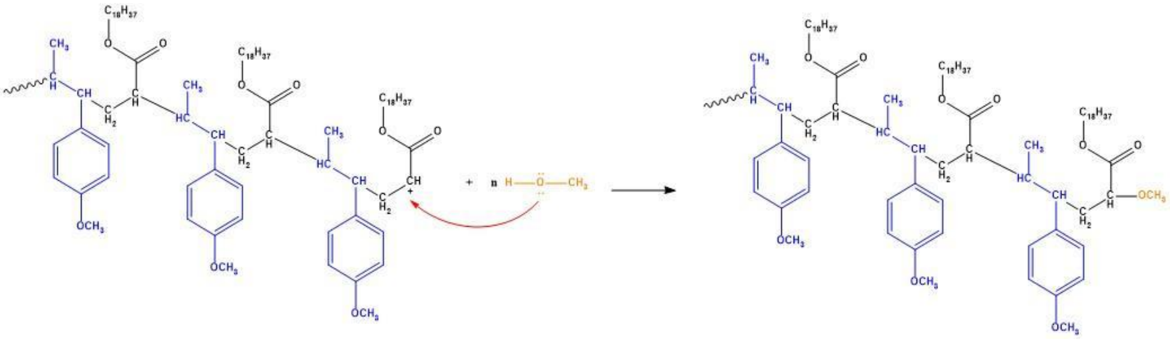

Figure 5. Possibility of Termination of Copoly(Anethole-SA)

Table 1. Synthesis Copoly(Anethole-SA)

Copoly (Anethole-SA)	Color	Starting Mass (g)		Product Mass (g)	Yield
		Anethole	SA		
2%	White	2	0.04	1.83	90 %
4%	Yellowish-white	2	0.08	1.87	90 %
6%	Yellowish	2	0.12	1.94	91 %

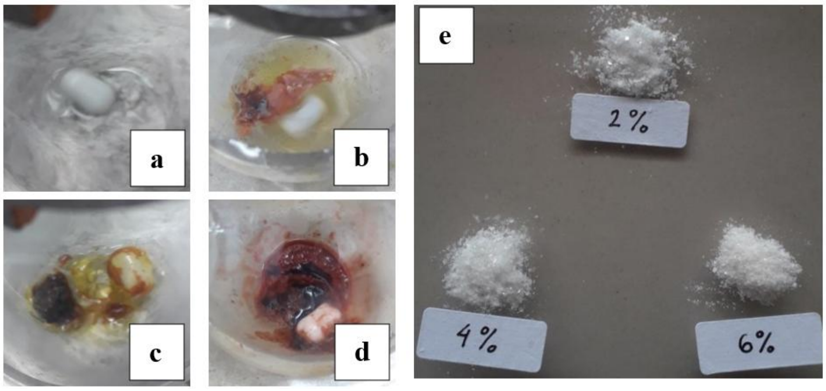

Figure 6. Copolymerization Stages and Results (a) Homogenization Process, (b) Initiation, (c) Propagation, (d) Termination, (e) Physical Form of Copoly 2%, 4% and 6%

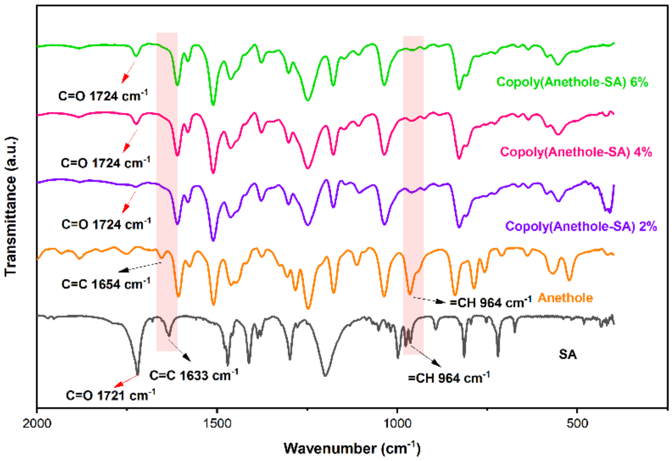

Figure 7. FTIR Spectrum of Anethole, SA and Copoly(Anethole-SA)

Analysis of Functional Groups Copoly(Anethole-SA)

Functional group analysis using FTIR obtained the spectrum shown in **Figure 7**. The vinyl groups (C=C stretching) on Anethole and SA at wavenumbers 1654 cm^{-1} and 1633 cm$^{-1,}$ respectively, were not found in copoly 2%, 4%, and 6%. This confirms that the copoly was successfully synthesized, characterized by the loss of the vinyl double bond (C=C) in Anethole and SA because it successfully added to a single bond (C-C).

Based on this study, the C=O stretching groups of SA appeared at 1721 cm^{-1}, and there was a shift in the copolymer at 1724 cm^{-1}. The C-O-C stretching group of SA appeared at wave numbers 1199 cm$^{-1,}$ which showed sp^3-O bonds. Meanwhile, anethole was present at wavenumbers 1176 cm^{-1}, 1248 cm^{-1}, indicating sp^2-O bonds, and 1036, 1112 cm$^{-1,}$ which showed sp^3-O bonds. The C-O-C stretching group on copolymer appeared at 1035 cm^{-1}, 1107 cm$^{-1,}$ showing sp^3-O bond, and 1177 cm^{-1}, 1248 cm^{-1} displaying the sp^2-O bond, while the possibility of -OCH$_3$ group termination in copoly appeared at wavenumber 1146 cm^{-1}.

Analysis using ^{1}H-NMR showed by proton signals contained in anethole, SA, and copoly compounds with CDCl$_3$ solvent. The success of copolymerization reaction is confirmed by the loss of the vinyl group proton signal in the copoly spectrum. Comparison of the ^{1}H-NMR spectra is illustrated in **Figure 8**.

^{1}H-NMR signal between Anethole, SA, and copoly on the proton copoly signal did not show any chemical signal shift in the range of 5,8–6,39 ppm, which is the chemical shift range of the vinyl group on Anethole and SA. This proved that the double bond in Anethole and SA was converted into a single bond (C-C).

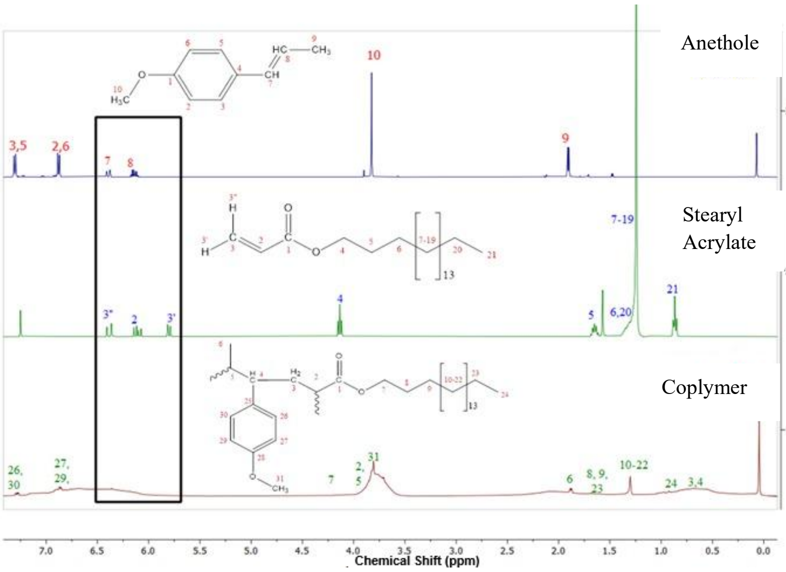

Figure 8. ^{1}H-NMR Spectrum of Anethole, SA and Copoly(Anethole-SA)

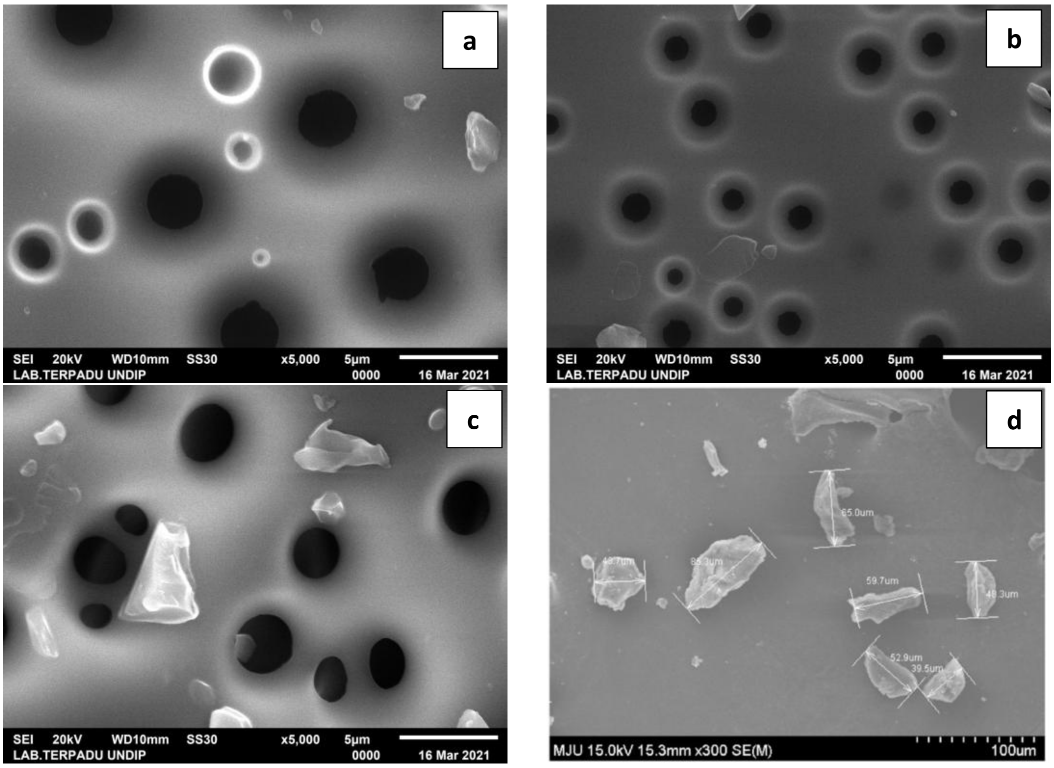

Figure 9. SEM Images at 5000x Magnification Showing Morphology of Copoly(Anethole-SA) at (a) 2%, (b) 4%, and (c) 6% (d) Polyanethole [5]

Characterization Copoly(Anethole-SA): Intrinsic Viscosity, Morphology, and Atomic Composition

The determination of the intrinsic viscosity of the copoly solution was carried out by measuring the viscosity of the copolymer solution using an Ostwald viscosimeter based on flow time in chloroform solvent. Furthermore, the intercept or intrinsic viscosity (η_i) of 2%, 4% and 6% coploy were 26.891, 41.093, and 55.336, respectively. Based on the equation of Mark–Houwink Sakurada, the intrinsic viscosity is directly proportional to molecular mass [17]. In this study, the molecular mass increased with the addition of SA. The molecular mass (Mv) of the copolymer was not determined because the value of the polymer-solvent system constant (K) and the soluble polymer constant of a solvent (a) were unknown.

The morphology of copoly(anethole-SA), characterized by SEM, is shown in **Figure 9**. Based on the study by Appiah et al (2018), the morphology of polyanethole (**Figure 9d**) is shown to have no cavities and is shaped like grains. The addition of SA resulted in the surface morphology of the copoly being hollow [5]. This is shown by Jang and Kim (2000), who stated that SA is shaped like a sponge. Furthermore, the addition of SA could form cavities in the copolymer, ensuring that it provides space for the adsorption process [8,18]. According to SEM results, it was observed that the cavities in copoly 4% were smaller, and the adsorption process produced by copoly was smaller than copoly 2% and 6%.

The addition of SA also affected the copoly constituents produced. This is shown in the EDX analysis based on a percentage of copolymer atoms, which is shown in **Table 2**. Based on the calculation of a percentage copolymer atoms, there was no significant change in the C and O elements when it was assumed that all anethole and SA had finished reacting.

TGA analysis performed at a one-step heating rate indicated that copoly underwent a single degradation reaction. The degradation temperature of copoly is presented in **Figure 10** and **Table 3**. The addition of SA caused the degradation temperature of copoly to increase. This is because the long carbon chain in SA causes copoly to have many branched chains, making it more difficult to degrade.

According to the results of this study, weak bonds break first [19]. The bond between the anethole group and SA in the copolymer formed a weak bond, ensuring that it breaks first. Furthermore, the amount of mass loss is strongly influenced by the -COOR group on SA [20].

Table 2. Atomic Composition of Copoly(Anethole-SA) with EDX

Elements	% Atom Copoly (Anethole-SA)					
	2%		4%		6%	
	Result	Theory	Result	Theory	Result	Theory
C	90.12	90.92	90.81	90.93	89.91	90.93
O	9.79	9.08	9.09	9.07	10.05	9.06

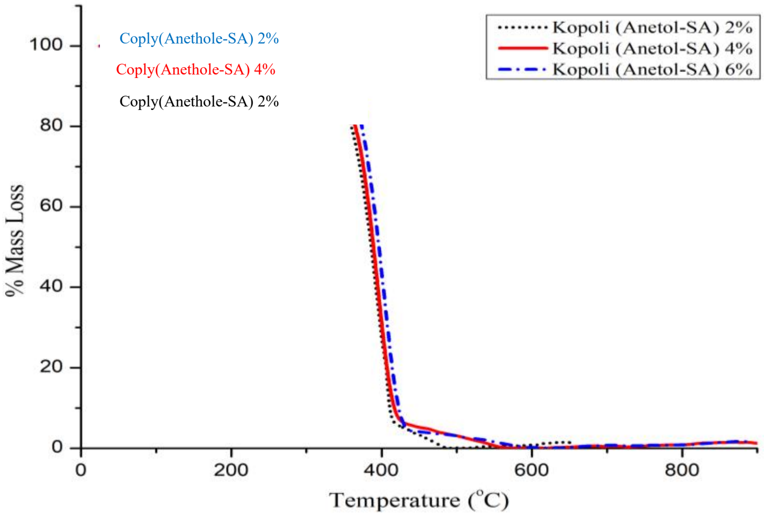

Figure 10. TGA Curve of Copoly(Anethole-SA)

Table 3. Degradation Temperature of Copoly(Anethole-SA)

Copoly (Anethole-SA)	T_i (°C)	T_f (°C)	Mass Loss
2%	363	441	94.67 %
4%	360	425	95.85 %
6%	357	416	97.38 %

Application of Copoly (Anethole-SA)

The adsorption process of phenol with copoly was conducted at room temperature with a time variation of 1 to 5 hours using an initial concentration of 10 ppm phenol. **Table 4** shows that the longer the contact time of the adsorption process, the greater the amount of phenol adsorbed, indicating an increase in adsorption capacity. This is because the longer the contact time, the more interactions are formed [21] between phenol and the copoly adsorbent. According to Kiswandono (2010) and Yang et al. (2016), the interactions that occur between the copoly and phenol are $\pi - \pi$ interactions and hydrogen bonds, as shown in **Figure 11** [21,22].

The aquadest solvent in the phenol solution caused hydrogen bonding competition between the copolymers and the solvent molecules. Therefore, it was necessary to optimize the pH variations to obtain maximum adsorption capacity. The adsorption capacity of 2%, 4%, and 6% copoly after 5 hours was 2.18, 2.11, and 2.22 mg/g, respectively.

At less than 5 hours, all the phenol had not been optimally adsorbed because the required contact time was insufficient. As the contact time increased, the adsorption rate increased, ensuring that the percentage of phenol adsorbed was greater [21]. Meanwhile, the variation in SA affected the phenol adsorption process based on the cavities observed in the copoly morphology. Based on SEM characterization in **Figure 9**, it was observed that the cavity in copoly 4% was smaller, indicating that the adsorption results were also smaller. This is because the larger of the cavity, the more adsorbate compounds are adsorbed [23,24].

In this study, the 6% copoly produced the largest adsorption capacity. This is because the cavity in the morphology was larger, and the functional groups present in the material can bind strongly to the adsorbed phenol, showing that the larger the cavity, the more phenol is adsorbed.

Table 4. Capacity and Percentage of Adsorbed Amount of Phenol

Time (hour)	Percentage Adsorption (%)			Adsorption Capacity(mg/g)		
	2%	4%	6%	2%	4%	6%
0	0	0	0	0	0	0
1	18.759	21.645	14.430	0.469	0.541	0.361
2	31.746	33.910	26.695	0.794	0.848	0.668
3	49.062	51.226	40.404	1.228	1.282	1.011
4	56.998	81.529	67.821	1.427	2.041	1.697
5	87.301	84.415	88.744	2.185	2.113	2.221

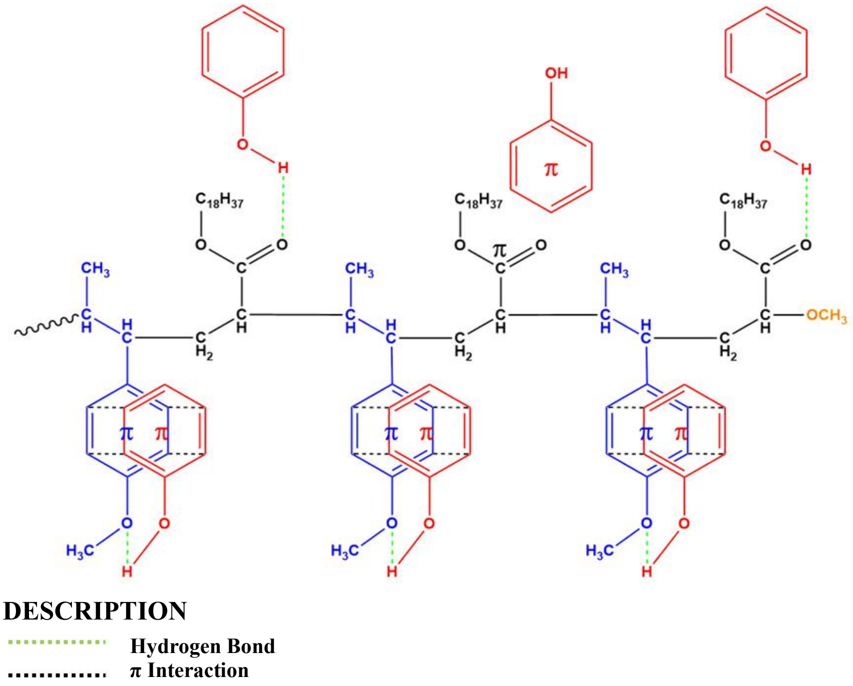

Figure 11. π Interaction and Hydrogen Bonds between Copoly(Anethole-SA)

4. CONCLUSIONS

In conclusion, the copoly has been successfully synthesized by cationic copolymerization without solvent using $BF_3O(C_2H_5)_2$ initiator under a nitrogen atmosphere. The synthesis was confirmed by FTIR analysis, which showed characteristic vinyl group peaks at 1633–1654 cm^{-1} (C=C stretching), 964–998 cm^{-1} (=CH vinyl bending), and 3025–3095 cm^{-1} (=CH vinyl stretching). Additionally, the disappearance of the vinyl proton signals at δH 5.80–6.39 in the ^{1}H-NMR spectrum further supports successful polymer formation. The addition of the percentage by weight of SA in the synthesis of copoly affects the intrinsic viscosity of copoly, which increases for 2%, 4%, and 6% variations by 26.891, 41.093, and 55.336, respectively. Furthermore, the morphology of copoly showed a hollow surface, but variation of copoly 4% had smaller cavities compared to the 2% and 6% copoly. The more the addition of SA, the degradation temperature of copoly will increase. The Copoly can be applied as an adsorbent for phenol compounds, with the adsorption capacity results for the 2%, 4% and 6% copoly not showing a significant difference. The percentage adsorption of the 2%, 4%, and 6% copoly toward phenol waste is 87.30%, 84.42%, and 88.74%, respectively. Meanwhile, the adsorption capacities for 5 consecutive hours are 2.185 mg/g, 2.113 mg/g, and 2.221 mg/g.

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